

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/075,019 Confirmation No. 7488  
Applicant : Steven Dietz et al.  
Filed : February 12, 2002  
TC/A.U. : 1732  
Examiner : Nguyen T. Ha  
Docket No. : 1867-00203  
Customer No.: 23505  
Title: Mesoporous Carbons and Polymers from Hydroxylated Benzenes

**RESPONSE TO OFFICE ACTION DATED MARCH 21, 2006**

Date: June 12, 2006

Mail Stop Amendment  
Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

**AMENDMENT AND  
RESPONSE TO OFFICE ACTION OF MARCH 21, 2006**

Sir:

This paper is filed in response to the Office Action dated March 21, 2006. The Examiner is requested to enter the following amendments and consider the accompanying remarks. Reconsideration and allowance are respectfully requested.

**Amendments to the Specification** begin on page 2 of this paper.

**Amendments to the Claims** are reflected in the listing of claims which begins on page 5 of this paper.

**Remarks** begin on page 10 of this paper.

**Amendments to the Specification:**

Please replace paragraph [0016] with the following:

[0016] In order to introduce larger pores into polymers, and thus increase their porosity, several groups have prepared polymeric gels, such as RF gels, in liquid emulsions. The term "gel" means only that a process such as polymerization has occurred in a liquid mixture causing the mixture to become more viscous or, in some cases, to become a solid. Such RF gels usually have a mixture of pore sizes, and both the polymers and the carbons formed therefrom by pyrolysis are mostly macroporous (i.e., having diameters greater than 50 nm) rather than mesoporous (LeMay et al. 1990, Even and Gregory 1994). An alternative approach uses aerogels, which are gels dried by supercritical fluid processing. Supercritical fluid processing is known to minimize shrinkage and pore collapse on drying, but is a relatively expensive processing ~~technique~~ techniques.

Please replace paragraph [0041] with the following:

[0041] The specific surface area and the pore size distribution of carbons prepared in this manner were analyzed by nitrogen adsorption on a Micromeritics Gemini instrument. Figure 3 shows the nitrogen isotherm of one of the samples. The isotherm is a Type 4 that is characteristic for mesoporous solids (Gregg and Sing 1982). Figure 4 shows a distinct mesopore centered at 12 nm, as calculated by the BJH method (Barrett et al. 1951). Typically greater than 80% of the pore volume is in the mesopore range.

Table 1. Formulation and properties of TDA's RF porous carbons.

| Weight Ratio<br>(water/resorcinol) | Mesopore<br>Sizes (nm) | Total Pore<br>Volume<br>(cm <sup>3</sup> /g) | Mesopore<br>Volume (cm <sup>3</sup> /g) | Total Surface<br>Area (m <sup>2</sup> /g) |
|------------------------------------|------------------------|--|---|---|
| 0.38                               | 12                     | 0.90   | 0.73                                    | 674                                       |
| 0.60                               | 18                     | 1.18   | 1.00                                    | 661                                       |
| 0.88                               | 28                     | 1.11   | 0.95                                    | 583                                       |

The properties of several RF derived carbons are shown in Table 1. If no excess water is added, carbons derived from these gels have low surface areas and no measurable mesopores. However, when excess water is added, the surface areas increase dramatically and the resulting carbons evidence mesopores. From this data, it is clear that the amount of excess water (i.e., water added in addition to that in the 37 weight percent formaldehyde solution) has a profound effect on the pore sizes. In fact, as shown in Figure 5, the mesopore size can be controlled in a very predictable ~~predicable~~ and linear manner simply by adjusting the excess water to resorcinol weight ratio.

Please replace paragraph [0046] with the following:

[0046] Irrespective of the exact mechanism of mesopore formation, in the preferred embodiments of the present invention, controlling the amount of additional water can be used to control the pore size in the resulting polymers and carbons. The preferred embodiments of the present invention therefore provide an inexpensive route to high surface area mesoporous polymers by avoiding the need for surfactants and for supercritical fluid extraction. However, nothing in the preferred embodiments of the present invention precludes the use of surfactants or supercritical fluid extraction and the present invention might also be practiced successfully using either or both of these. This technique also allows control over the pore size of the resultant carbon in the mesopore range, a result that has not been possible without the use of surfactants.

**Amendments to the Claims:**

This listing of claims will replace all prior versions, and listings, of claims in the application.

1-39. (canceled)

40. (currently amended) A method for preparing a mesoporous polymer, comprising the steps of:

- (a) providing a solution containing a solvent and one or more polymerizable organic compounds whose polymerization rate depends on pH;
- (b) controlling the average polymer particle size primarily by controlling the pH of the solution; and
- (c) manipulating the pore size in the polymer primarily by adjusting the solvent concentration.

41. (original) The method according to claim 40, further including the step of drying the porous polymer to produce a dried porous polymer.

42. (currently amended) A method for preparing a mesoporous carbon, comprising the steps of:

- (a) providing a solution containing a solvent and one or more polymerizable organic compounds whose polymerization rate depends on pH;
- (b) controlling the average polymer particle size primarily by controlling the pH of the solution;
- (c) manipulating the pore size in the polymer by adjusting the solvent concentration; and
- (d) pyrolyzing the porous polymer to form a porous carbon.

43. (original) The method according to claim 42, further including the step of activating the porous carbon so as to increase its capacitance when used with an electrolyte.

44. (original) The method according to claim 42, further including the step of activating the porous carbon, wherein the activation is carried out at between 500°C and 1200°C.

45. (original) The method according to claim 42, further including the step of controlling the composition of the solution so as to produce a mesoporous carbon having a pore size between 2 and 50 nm.

46. (original) The method according to claim 42, further including the step of controlling the composition of the solution so as to produce a mesoporous carbon having a pore size between 10 and 28 nm.

47. (withdrawn) A capacitor, comprising:

at least two electrodes, at least one of said electrodes comprising a mesoporous carbon material produced according to claim 42; and  
an electrolyte in contact with at least one of said electrodes.

48. (withdrawn) The capacitor according to claim 47 wherein the electrolyte is a non-aqueous electrolyte.

49. (withdrawn) The capacitor according to claim 47 wherein the electrolyte is an aqueous electrolyte.

50. (withdrawn) The capacitor according to claim 47 wherein the carbon material is monolithic.

51. (withdrawn) A mesoporous carbon prepared according to claim 42 with at least one dimension greater than 2 mm, a surface area between 200 and 2000 m<sup>2</sup>/g, a density greater than 0.5 g/cc, and a pore size greater than 10 nm.

52. (withdrawn) The mesoporous carbon according to claim 51 having a conductivity of at least 10 Scm<sup>-1</sup>.

53-69. (canceled)

70. (new) The method according to claim 40, further including the step of controlling the composition of the solution so as to produce a mesoporous polymer having a pore size between 2 and 50 nm.

71. (new) The method according to claim 40, further including the step of controlling the composition of the solution so as to produce a mesoporous polymer having a pore size between 10 and 28 nm.

72. (new) The method according to claim 40, wherein the pH of the solution is less than 6.5.

73. (new) The method according to claim 40 wherein said solution is essentially free of catalyst.

74. (new) The method according to claim 40 wherein said solution is essentially free of surfactant.

75. (new) The method according to claim 40 wherein the pH of the solution is less than 6.5 and the solution is essentially free of surfactant.

76. (new) The method according to claim 42, wherein the pH of the solution is less than 6.5.

77. (new) The method according to claim 42 wherein the solution is essentially free of catalyst.

78. (new) The method according to claim 42 wherein the solution is essentially free of surfactant.

79. (new) The method according to claim 42 wherein the pH of the solution is less than 6.5 and the solution is essentially free of surfactant.

80. (new) A method for preparing a mesoporous polymer, comprising the steps of:

a) providing an aqueous acidic solution containing one or more polymerizable organic compounds; and

b) polymerizing the polymerizable organic compounds;

wherein step b) includes controlling the average polymer particle size primarily by controlling the pH of the solution and controlling the pore size in the polymer primarily by adjusting the ratio of water to polymerizable organic compounds.

81. (new) The method according to claim 80 wherein one of the polymerizable organic compounds comprises a hydroxylated benzene.

82. (new) The method according to claim 80, further including the step of drying the porous polymer to produce a dried porous polymer.

83. (new) The method according to claim 80 wherein step b) is carried out so as to produce a mesoporous polymer having a pore size between 2 and 50 nm.

84. (new) The method according to claim 80 wherein step b) is carried out so as to produce a mesoporous polymer having a pore size between 10 and 28 nm.

85. (new) A method for preparing a mesoporous carbon, comprising the steps of:

a) providing an aqueous acidic solution containing one or more polymerizable organic compounds;

b) polymerizing the polymerizable organic compounds;

wherein step b) includes controlling the average polymer particle size primarily by controlling the pH of the solution and controlling the pore size in the polymer primarily by adjusting the ratio of water to polymerizable organic compounds; and



c) pyrolyzing the porous polymer to form a porous carbon.

86. (new) The method according to claim 85 wherein one of the polymerizable organic compounds comprises a hydroxylated benzene.

87. (new) The method according to claim 85, further including the step of activating the porous carbon so as to increase its capacitance when used with an electrolyte.

88. (new) The method according to claim 85, further including the step of activating the porous carbon, wherein the activation is carried out at between 500°C and 1200°C.

89. (new) The method according to claim 85, further including the step of controlling the composition of the solution so as to produce a mesoporous carbon having a pore size between 2 and 50 nm.

90. (new) The method according to claim 85, further including the step of controlling the composition of the solution so as to produce a mesoporous carbon having a pore size between 10 and 28 nm.

### **REMARKS**

Applicants acknowledge receipt of the Office Action dated March 21, 2006.

#### **Status of the Claims:**

Claims 1-39 are canceled.

Claims 47-52 are withdrawn.

Claims 53-69 are canceled.

Claims 40-46 and 70-88 are pending.

Claims 70-88 are new.

#### **Rejections Under 35 USC §102(e)**

In the Office Action mailed on March 21, 2006, claims 40-52 are rejected under 35 USC § 102 (e) as being anticipated by Bell *et al.*, U.S. Patent No.6,297,293 (“*Bell*”). For anticipation under 35 USC § 102, the reference must teach every aspect of the claimed invention either explicitly or impliedly. Any feature not directly taught must be inherently present (MPEP 706.02 IV). When a U.S. patent, a published U.S. patent application, or an international application publication is used to reject claims under 35 U.S.C. 102 (e), the disclosure relied on in the rejection must be present in the issued patent or application publication (MPEP 2136.02 II).

In support of the rejection of the present claims, the Examiner states that:

“...[R]egarding claim 40, *Bell* discloses a method for preparing a mesoporous polymer comprising the steps of: providing a solution containing one or more polymerizable compounds (column 17, lines 2-5 and column 6, lines 11-27) whose polymerization rate depends on pH; it is inherent that the polymerizable organic compound and a surfactant as taught by *Bell* having the pH level.”

This is somewhat unclear, but it appears that the Examiner intends to assert that it is inherent that the polymerization rate of *Bell* depends on the pH. In response, Applicants respectfully point out that *Bell* actually utilizes a catalyst to control the polymerization rate, and notes that only basic conditions were effective (*Bell*, Column 6, Lines 40-41 and 46-47). Applicants submit that use of a reaction that may be *affected by* pH, does not anticipate a method that includes the *use of pH* to control the reaction, as presently claimed.

The Examiner further rejects claim 40 because, he states, *Bell* discloses “controlling the average polymer particle size primarily by controlling the pH” and “manipulating the pore size in the polymer primarily by adjusting the solvent concentration.” Applicants respectfully disagree.

*Bell* does not disclose either that the average polymer particle size can be controlled primarily by controlling the pH or that the pore size in the polymer can be manipulated primarily by adjusting the solvent concentration. *Bell*’s surfactant templating synthesis controls the pore size by controlling the amount of a necessary additional solute (i.e. the surfactant) in the solution. With respect to pore size, *Bell* specifically states that *pore size is controlled through adjustment of the amount of surfactant in solution*. *Bell*’s surfactant forms micelles about which polymerization occurs. “The size of the micelles controls the pore size of the material” (Column 4, Lines 62-63).

*Bell* also found that “a control sample with no surfactant contained only micropores (Column 7, Lines 1-5). Further, *Bell* states that “as expected the polymer prepared without any AOT (a surfactant) did not show any pores in the mesopore range” (Column 10, Lines 50-53). Thus, nowhere does *Bell* teach or suggest a method whereby the pore size in the polymer is manipulated primarily by adjusting the solvent concentration, as recited in present claim 40. Furthermore, the present invention is distinguishable over *Bell* for its recitation that that the average polymer particle size is controlled primarily by the pH. Nowhere in *Bell* is this disclosed or suggested.

Thus, the present invention is distinguishable over *Bell* at least in that it does not require the use of surfactants, which can be quite expensive, and that it provides a product in which pore size and particle size can be controlled by novel means.

In light of the fact that the cited reference does not teach every aspect of the claimed invention, either explicitly or impliedly, Applicants request that the 35 U.S.C. 102(e) rejection be reconsidered and withdrawn, and that claim 40 be allowed. Because claims 41 and 70-75 depend from claim 40, they are also submitted to be patentable and unanticipated by *Bell*.

For much the same reasons, Applicants submit that claim 42 is not anticipated by *Bell*. Specifically, *Bell* does not teach or disclose a polymer fabrication method in which the pore size in the polymer is controlled primarily by adjusting the solvent concentration, or that the average polymer particle size is controlled primarily by controlling the pH. Thus, Applicants request that

the rejection of claim 42 be withdrawn and that claims 42 and its dependent claims 43-46 and 76-79 be allowed.

**New Claims:**

Claims 70-88 are new.

Claims 70-75 depend from independent claim 40. Since independent claim 40 is submitted to be allowable for the reasons set out above, Applicants submit that dependent claims 70-75 are also allowable.

Claims 76-79 depend from independent claim 42. Since independent claim 42 is submitted to be allowable for the reasons set out above, Applicants submit that dependent claims 76-79 are also allowable.

In addition, claims 72, 75, 76 and 79 each require that the pH of the solution be less than 6.5 (Supported in original claims). This recitation renders claims 72, 75, 76 and 79 clearly distinguishable over and not obvious in view of *Bell*, which teaches the use of a basic solution to achieve the desired results.

Similarly, claims 73 and 77 both require that the polymerization be carried out in a solution that is essentially free of catalyst. (Supported at paragraph [0038]). This recitation renders claims 73 and 77 clearly distinguishable over and not obvious in view of *Bell*, which teaches that a basic catalyst is required to achieve the desired results.

Likewise, claims 74, 75, 78, and 79 each require that the polymerization solution be essentially free of surfactant. This recitation renders claims 74, 75, 78, and 79 patentable over *Bell*, which teaches the use of a surfactant-containing solution.

Claim 80 is a new independent claim that recites an embodiment to which the Applicants are entitled (reference specification paragraphs [0025], [0037], [0041], for example). Claim 80 is narrower in scope than claim 40, which is submitted to be allowable. Hence, new independent claim 80 is submitted to be allowable. New claims 81-83 depend from new independent claim 80 and are also allowable for the reasons set out above.

Like claim 80, claim 84 is a new independent claim that recites an embodiment to which the Applicants are entitled (reference specification paragraphs [0025], [0037], [0041], for example), and that is more limited than claim 42, which is submitted to be allowable. Hence,

new independent claim 84 is submitted to be allowable. New claims 85-88 depend from new independent claim 84 and are also allowable for the reasons set out above.

In summary, new claims 70-88, which relate to the same subject matter as the previously elected claims, are each drawn to an embodiment to which the Applicants are entitled and are distinguishable over the art of record. Thus, Applicants request the Examiner's allowance of the new claims.

**Additional Remarks:**

The Examiner is asked to note that *Bell* (U.S. Patent No.6,297,293) and the present application are co-owned, and that the present application was filed within one year of the issue date of *Bell*.

**Conclusion:**

Applicants respectfully submit that claims 40-46 and 70-88, as herein presented, are in condition for allowance. No new matter undisclosed in the specification is added by way of the herein submitted new claims 70-88. Reconsideration of the application in light of the foregoing remarks and withdrawal of the 35 U.S.C. 102(e) rejections made in the Office Action dated March 21, 2006, to claims 40-46, are respectfully requested.

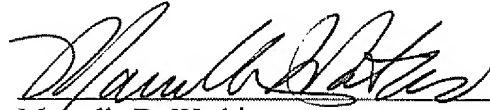
In the course of the foregoing discussions, Applicants may have at times referred to claim limitations in shorthand fashion, or may have focused on a particular claim element. This discussion should not be interpreted to mean that the other limitations can be ignored or dismissed. The claims must be viewed as a whole, and each limitation of the claims must be considered when determining the patentability of the claims. Moreover, it should be understood that there may be other distinctions between the claims and the prior art that have yet to be raised, but which may be raised in the future.

If any item in the Office Action has been overlooked or is deemed to be incompletely addressed, Applicants respectfully request the opportunity to respond. If any fees are inadvertently omitted or if any additional fees are required or have been overpaid, please appropriately charge or credit those fees to Conley Rose, P.C. Deposit Account Number 03-2769.

App. No : 10/075,019  
Amendment Dated June 21, 2006  
Reply to Office Action of March 21, 2006

If the Examiner has any questions or comments, or otherwise feels it would be advantageous, he is encouraged to telephone the undersigned at (713) 238-8043.

Respectfully submitted,

A handwritten signature in cursive script, appearing to read 'Marcella D. Watkins', written over a horizontal line.

Marcella D. Watkins

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